



Research Article

An Integrated Coagulation-Sedimentation, O₃-AOP, and Membrane Bioreactor (MBR) Hybrid System for The Degradation of Recalcitrant Organic Pollutants in Simulated Pharmaceutical and Resin Synthesis Wastewater

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Abstract

The treatment of high-strength wastewater from pharmaceutical and resin synthesis processes, which contain recalcitrant organic pollutants and emerging contaminants, remains a major environmental challenge because of their high chemical oxygen demand (COD), low biodegradability, and toxicity. In this study, the performance of a laboratory-scale hybrid system integrating coagulation–sedimentation, ozone-based advanced oxidation (O₃-AOPs), and a membrane bioreactor (MBR) for the removal of COD, phenol, and paraben from three simulated wastewater scenarios was evaluated. The system achieved overall COD removal efficiencies exceeding 95% across all the scenarios, with the highest removal (97.4%) observed in high-strength pharmaceutical wastewater (influent COD concentration of 4,398–4,750 mg L⁻¹). Phenol removal exceeded 98% in all the cases, while target parabens were reduced to below the analytical detection limit and were subsequently reported as nondetectable. Spatial attenuation profiling along the ozone contact column suggested a dual-mechanism behavior. The rapid pollutant attenuation observed in the lower section was consistent with radical-driven oxidation, whereas the enhanced removal in the upper section may indicate the contribution of additional processes, including possible physical stripping of volatile intermediates. The O₃-AOP stage served as an effective protective barrier, reducing phenol concentrations to below the biological inhibition threshold before the wastewater entered the MBR. The findings demonstrate that the triple-integrated hybrid system provides an effective multiple-barrier treatment approach for complex industrial wastewater, offering high pollutant removal efficiency and substantially reducing the concentration of target cytotoxic and potentially endocrine-disrupting compounds.

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Introduction

The rapid expansion of the pharmaceutical and polymer (resin) manufacturing industries has generated substantial economic benefits while simultaneously

producing large volumes of complex industrial wastewater. Effluents from these sectors contain high concentrations of recalcitrant organic pollutants and emerging contaminants (ECs), including pharmaceutical

residues, organic solvents, and residual monomers (Asheghmoalla and Mehrvar, 2024; Lastre-Acosta et al., 2020). A defining feature of such wastewaters is their extremely high chemical oxygen demand (COD) coupled with very low biodegradability, which is typically reflected in BOD₅/COD ratios below 0.3. The presence of complex aromatic structures and toxic chemical constituents renders these effluents recalcitrant to biodegradation and poses significant ecological risks to receiving water bodies (Mascolo et al., 2010).

Conventional biological treatment processes frequently underperform when applied to these waste streams. High organic loading and the presence of toxic compounds readily inhibit or destroy microbial communities, leading to process instability and failure of biological systems (Laera et al., 2012). To overcome these limitations, advanced oxidation processes (AOPs), particularly ozone-based AOPs (O₃-AOPs), have emerged as effective chemical pretreatment options. In aqueous solution, ozone can directly oxidize target pollutants or decompose them to generate highly reactive hydroxyl radicals ($\cdot\text{OH}$) (Hansen et al., 2016). These non-selective radicals effectively cleave stable aromatic rings, converting recalcitrant macromolecules into simpler, more biodegradable aliphatic intermediates. Consequently, O₃-AOP pretreatment substantially reduces toxicity and enhances the biodegradability of pharmaceutical and resin wastewaters prior to biological treatment (Prado et al., 2017).

Despite its oxidative power, the standalone application of O₃-AOP for extensive degradation is rarely economically viable because of excessive chemical and energy consumption (Asheghmoalla and Mehrvar, 2024). Conversely, membrane bioreactors (MBRs) deliver superior effluent quality through effective biomass retention and solid–liquid separation. However, MBR systems are highly susceptible to membrane fouling, which is exacerbated by the accumulation of recalcitrant organic matter and the excessive secretion of extracellular polymeric substances (EPSs) under toxic stress (Namdeti et al., 2025).

The integration of coagulation–sedimentation, O₃-AOPs, and MBR into a single hybrid configuration offers a promising complementary solution. In this arrangement, coagulation–sedimentation removes suspended solids and colloidal matter, whereas O₃-AOPs function as a critical pretreatment barrier that detoxifies the influent and fragments large, inhibitory molecules before they reach the MBR (Laera et al., 2012). This upstream oxidation step may reduce the organic loading delivered to the downstream biological stage and facilitate subsequent biological treatment. The partially oxidized intermediates are subsequently mineralized by the microbial community within the

MBR, and the ultrafiltration membrane ensures complete retention of the biomass and residual solids, enabling the hybrid system to meet stringent discharge standards (Quyen et al., 2016).

Although hybrid AOP–MBR systems have been investigated for various industrial effluents, most existing studies have focused on either single-process enhancements or binary combinations (e.g., ozonation followed by MBR) applied to relatively moderate-strength wastewaters. Comprehensive evaluations of a triple-integrated coagulation–sedimentation + O₃-AOPs + MBR configuration for treating high-strength pharmaceutical and resin synthesis wastewaters—particularly under multiple contamination scenarios with detailed spatial kinetic profiling along the ozone contactor—are still limited. Furthermore, the mechanisms underlying the attenuation pathways for high-concentration phenolic compounds and trace endocrine-disrupting compounds, together with preliminary insights into potential toxicity reduction based on target pollutant elimination, remain insufficiently addressed in the literature.

The present study was therefore undertaken to develop and rigorously evaluate a laboratory-scale hybrid coagulation–sedimentation + O₃-AOPs + MBR system for the treatment of simulated pharmaceutical and resin-synthesis wastewater. The specific objectives were to (i) quantify overall removal efficiency for bulk organic matter (COD) and target micropollutants (phenol and paraben) across representative wastewater scenarios; (ii) evaluate the spatial attenuation behavior of pollutant removal along the ozone contact column; (iii) evaluate the protective role of O₃-AOPs in lowering phenol concentrations below the biological inhibition threshold prior to biological treatment; and (iv) assess pollutant removal performance within the downstream MBR following ozone pretreatment.

Materials and methods

1) Laboratory-scale hybrid system configuration

An integrated hybrid system consisting of coagulation–sedimentation, ozone-based advanced oxidation (O₃-AOP), and MBR was designed and constructed at the laboratory scale. The entire setup was compactly assembled on a single stainless-steel support frame measuring 181 cm (length) × 137 cm (width) × 56 cm (height) to optimize space utilization and facilitate operation and maintenance (Figure 1).

Coagulation–sedimentation pretreatment was performed using polyaluminum chloride (PAC) as the primary coagulant at an optimal dosage of 100 mg L⁻¹, with the wastewater pH adjusted to 7.0. The coagulation procedure consisted of rapid mixing at 150 rpm for 2 min, followed by slow flocculation at 40 rpm for 20 min and a sedimentation period of 30 min before the clarified supernatant was collected.



Figure 1 Schematic and experimental setup of the integrated laboratory-scale coagulation–sedimentation/ O_3 -AOP/MBR hybrid system for treating simulated pharmaceutical and resin synthesis wastewater.

Ozonation was performed in a transparent polyacrylic column with a total working volume of 20 L, an internal height of 150 cm, and an internal diameter of 100 mm. Ozone gas was generated on-site by an industrial Nion N_3 ozone generator with a rated ozone production rate of 3.0 g h^{-1} . The ozone-containing feed gas was continuously sparged into the bottom of the contact column through a fine-bubble diffuser disc ($\varnothing$ 100 mm) in countercurrent flow mode at a constant gas flow rate of 1.0 L min^{-1} (equivalent to $0.06 \text{ m}^3 \text{ h}^{-1}$). On the basis of the rated ozone generation capacity and gas flow rate, the estimated inlet ozone concentration ($C_{g, \text{in}}$) was approximately 50.0 g m^{-3} (50.0 mg L^{-1}). On the basis of a design wastewater flow rate of 100 L d^{-1}

(4.17 L h^{-1}), the system was operated with an applied ozone dose (AOD) of approximately 720 mg L^{-1} . This high applied ozone dose provided a strong oxidative driving force for the degradation of organic matter and target micropollutants. Dissolved ozone and off-gas ozone concentrations were not monitored during the present study; therefore, the ozone transfer efficiency and actual ozone consumption could not be quantitatively determined. To prevent downstream biological inhibition, the ozonated effluent was subjected to forced air stripping for 15 min to remove residual dissolved ozone before being introduced into the biological reactor.

The membrane bioreactor unit was equipped with a hollow-fiber ultrafiltration membrane module made of polyvinylidene fluoride (PVDF) with a nominal pore size of 0.04 μm . The membrane module was submerged in the bioreactor tank and operated under constant-flux mode with periodic backwashing and relaxation cycles to control fouling. The hybrid system was operated in a continuous, closed-loop configuration. Raw wastewater first passes through a pH adjustment tank and then through coagulation and sedimentation to remove suspended solids and colloidal particles. The clarified effluent then entered the ozone contact column for advanced oxidation pretreatment. Finally, the ozonated wastewater flowed into the MBR unit for biological degradation and final solid–liquid separation.

Biological treatment was conducted in a submerged MBR with a working volume of 10.0 L, which was equipped with a hollow-fiber PVDF membrane module (Zenon ZW-1) with a nominal pore size of 0.04 μm and an effective membrane area of 0.047 m^2 . The reactor was operated at a constant permeate flux of 5.6–6.0 $\text{L m}^{-2} \text{h}^{-1}$, resulting in a continuous hydraulic retention time (HRT) of 6.0 h. Mixed liquor-suspended solids (MLSS) concentrations were maintained within the range of 6,000–8,000 mg L^{-1} under an average sludge retention time (SRT) of 30 d. Continuous aeration was supplied at a rate of 8.0 L min^{-1} for oxygen transfer and membrane scouring.

2) Simulated wastewater preparation and experimental scenarios

To ensure experimental consistency and reproducibility, simulated wastewaters were prepared using analytical-grade compounds. Glucose and peptone were used to construct the bulk organic matrix, while phenol and parabens were added as model target pollutants. Nutrient supplementation was provided using NH_4Cl and KH_2PO_4 to maintain an approximate COD:N:P ratio of 250:5:1 for biological treatment.

Three simulated wastewater scenarios were prepared to represent typical pollution profiles of the pharmaceutical and resin synthesis industries. All the experiments were conducted at a constant hydraulic loading rate of 100 L day^{-1} . The three scenarios were as follows:

- **Mixed pharmaceutical and resin wastewater:**

This wastewater was characterized by a COD of 2,500–3,000 mg L^{-1} , a phenol concentration of 400–500 mg L^{-1} , and a paraben concentration of 0.1–0.3 mg L^{-1} . Sampling was performed twice during steady-state continuous operation. To monitor the progressive biodegradation profile within the bioreactor, samples were collected at MBR treatment intervals of 1, 2, and 3 h, as well as from the raw influent, ozonated effluent, and the final steady-state permeate corresponding to the designed continuous HRT of 6.0 h.

- **High-strength pharmaceutical wastewater:**

This wastewater represents the most challenging condition, with a COD concentration ranging from 4,500–5,000 mg L^{-1} , a phenol concentration ranging from 100–200 mg L^{-1} , and trace paraben concentrations ranging from 0.01–0.05 mg L^{-1} . Sampling was conducted twice per scenario. Each run included one raw influent sample, three samples collected along the height of the ozone column (at 0.5 m, 0.75 m, and 1.0 m from the bottom), and one final effluent sample after the MBR.

- **Resin synthesis wastewater** featured the highest phenol toxicity (700–800 mg L^{-1}), moderate COD concentration (1,500–2,000 mg L^{-1}), and elevated paraben concentration (0.5–1 mg L^{-1}). This scenario was operated continuously for five days. Sampling was performed twice, with each run consisting of one raw influent sample, three samples along the ozone column height, and one MBR effluent sample.

3) Kinetic analysis and rate constant estimation

To evaluate the contributions of different mechanisms along the height of the ozone contact column, the apparent rate constants for oxidation and stripping were estimated using a spatial approach. Because the hydraulic retention time at each sampling height could not be precisely determined, the pseudo-first-order kinetic model was applied with respect to column height rather than reaction time. The following relationship (Eq. 1) was employed:

$$\ln\left(\frac{C_{\text{in}}}{C_{\text{out}}}\right) = k \cdot \Delta h \Rightarrow k = \frac{\ln(C_{\text{in}}/C_{\text{out}})}{\Delta h} \quad (\text{Eq. 1})$$

where C_{in} and C_{out} represent the COD concentrations entering and leaving a considered section of the column, respectively; Δh is the difference in height between the two sampling points (m); and k is the apparent rate constant with respect to the column height (m^{-1}).

The estimation was conducted in two steps corresponding to the two main zones of the column: Estimation of $k_{\text{oxidation}}$: The rate constant for the oxidation process was calculated using data from the lower section of the column (0–0.5 m). In this region, chemical oxidation was assumed to be the predominant mechanism, as the original organic compounds were relatively large and less susceptible to volatilization. Therefore, the apparent rate constant derived from this section was considered to primarily reflect the oxidation process. Assessment of the stripping contribution in the upper section: The apparent rate constant for the upper section of the column (0.5–1.0 m) was calculated using the same approach. By comparing these values with $k_{\text{oxidation}}$ obtained from the lower section, the additional contribution of physical stripping in the upper part of the column could be qualitatively evaluated. This comparison was based on the assumption that any significant increase

in the apparent rate constant beyond the oxidation-dominated value could be attributed, at least in part, to the stripping effect induced by continuous gas sparging.

This simplified spatial kinetic approach allowed estimation of the relative importance of chemical oxidation and physical stripping along the ozone contact column without requiring direct measurements of gas-phase compounds or precise local hydraulic retention times.

4) Analytical methods

The paraben concentration was quantified using a high-performance liquid chromatography (HPLC) system equipped with a UV-Vis/DAD detector and a reversed-phase C18 column (150 mm × 4.6 mm, 5 µm). The calibration curve was linear over the concentration range employed for analysis. Method detection limits (LODs) and quantification limits (LOQs) were established in accordance with the analytical laboratory validation protocol. Routine quality control procedures included blank analyses, replicate injections, and periodic calibration verification throughout the analytical process. To ensure the reliability and reproducibility of the experimental data, all treatment scenarios and analytical measurements were performed in duplicate. The pollutant concentrations and removal efficiencies presented in the tables and figures correspond to the average values obtained from parallel experimental runs.

Results and discussion

1) Overall treatment performance

The treatment efficiency of the hybrid coagulation–sedimentation/O₃-AOP/MBR system was evaluated by measuring the removal of key parameters from the influent to the final permeate after the membrane bioreactor. Among the three target parameters, paraben was completely removed during the O₃-AOP stage. Consequently, only COD and phenol remained as the primary parameters requiring further treatment and polishing by the ultrafiltration membrane in the MBR unit.

1.1) COD removal efficiency

The hybrid system demonstrated excellent resilience and treatment capacity under high organic loading conditions across all three wastewater scenarios (Figure 2a). The most challenging condition was high-strength pharmaceutical wastewater, with influent COD concentrations ranging from 4,398 to 4,750 mg L⁻¹. After passing through the entire hybrid system, the effluent COD concentration was reduced to 103–133 mg L⁻¹, corresponding to an overall removal efficiency of 97.4%. For the mixed pharmaceutical-resin wastewater, the COD concentration decreased from 2,750–3,310 mg L⁻¹ in the influent to 113–128 mg L⁻¹ in the final effluent, achieving a removal efficiency of 96.0%.

Notably, even the resin synthesis wastewater, which contained high concentrations of toxic phenolic compounds, was effectively treated, with the COD concentration reduced from 1,654–2,125 mg L⁻¹ to 67–94 mg L⁻¹ (removal efficiency of 95.7%). This observation aligns with those of multiple reviews and case studies showing that compared with single-process systems, hybrid or integrated systems combining biological and advanced oxidation or adsorption steps yield superior removal of recalcitrant organics under stress loading (Echevarría et al., 2019; Jasemizad et al., 2018). In pharmaceutical-dominated effluents, MBR-based hybrids coupled with AOPs or PAC-enhanced bioreactors have shown high COD removal efficiency and performance under high-loading conditions (Echevarría et al., 2019; Tran et al., 2014).

These high removal efficiencies (>95%) across all the scenarios highlight the complementary effect of the integrated system. O₃-AOP pretreatment plays a critical role by cleaving complex aromatic structures and recalcitrant organic molecules into simpler, more biodegradable aliphatic compounds (Možar et al., 2023). This molecular breakdown significantly reduced the organic shock load and toxicity to the microbial community in the MBR, allowing more extensive degradation of the remaining organic matter (Arabi et al., 2020; Kishor et al., 2021; Ni et al., 2024). In the subsequent MBR stage, with high biomass retention and effective solid–liquid separation, the effluent was further polished to consistently low COD levels. The final effluent COD concentrations in all three scenarios approached or met the stringent discharge limits specified in QCVN 40:2025/BTNMT (Column B), demonstrating the practical applicability of the hybrid system for treating highly polluted industrial wastewaters.

1.2) Phenol removal efficiency

The performance of the hybrid system for phenol removal is shown in Figure 2b. Although overall phenol removal exceeded 90% across all the scenarios, the system struggled to achieve very low effluent concentrations because of extremely high influent phenol levels, particularly in the resin synthesis wastewater. In the mixed wastewater scenario, the concentration of phenol was reduced from 477 mg L⁻¹ to 5 mg L⁻¹. In the high-strength pharmaceutical wastewater, the concentration of phenol decreased from 134 mg L⁻¹ to 2 mg L⁻¹. With the highest phenol concentration (865 mg L⁻¹), the concentration of the final effluent still substantially decreased to 13 mg L⁻¹.

These results confirm that the O₃-AOP stage effectively initiates phenol degradation via hydroxyl radical attack on the aromatic ring. However, the extensive degradation of phenol to meet the strictest discharge standards often requires the combined action of both advanced oxidation and biological

treatment. The MBR stage contributes to additional phenol reduction through the degradation of partially oxidized intermediates (Esteki et al., 2024). The relatively high residual phenol concentration in the resin wastewater effluent (13 mg L⁻¹) suggests that for waste streams with exceptionally high phenol concentrations, additional optimization, such as increased ozone dosage, longer hydraulic retention time in the MBR, or a tertiary polishing step, may be necessary to consistently comply with the most stringent regulatory limits.

The hybrid coagulation–sedimentation/O₃-AOP/MBR system was highly effective at treating complex, high-strength pharmaceutical and resin-synthesis wastewaters (Esteki et al., 2024). The combination of chemical oxidation for molecular cleavage and concentration reduction, along with biological degradation and membrane separation, enabled effective performance across a wide range of pollution profiles while maintaining stable effluent quality.

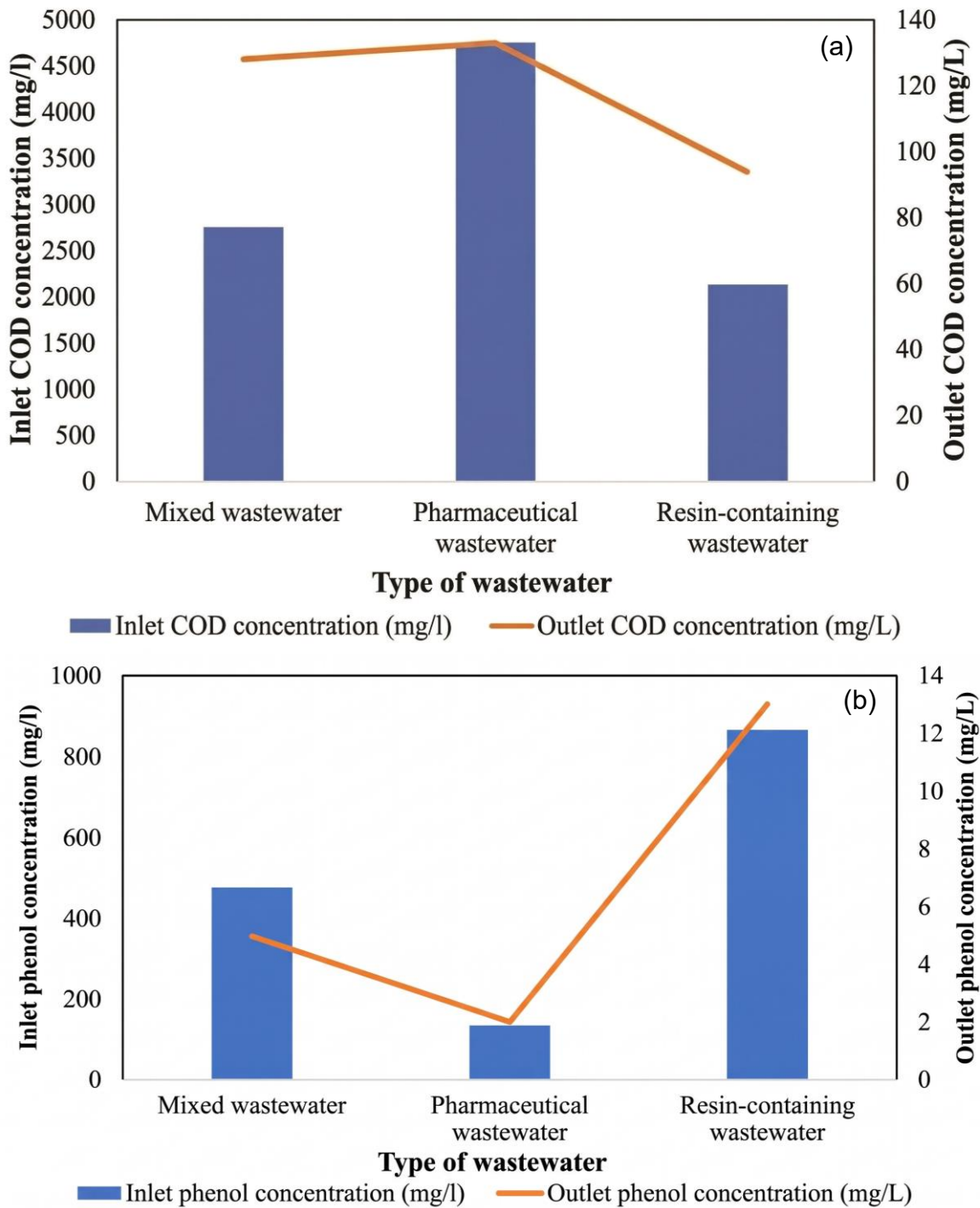


Figure 2 Spatial concentration profiles of COD and phenol along the vertical height of the ozone contact column under different simulated industrial wastewater scenarios.

1.3) Paraben removal efficiency

Among all the evaluated parameters, paraben had the highest removal efficiency in the hybrid system. As a representative micropollutant of pharmaceuticals and personal care products (PPCPs), parabens are classified as persistent organic compounds with recognized endocrine-disrupting potential. Influent paraben concentrations across the three wastewater scenarios ranged from 0.028 to 1.195 mg L⁻¹. The experimental results revealed that paraben concentrations were reduced to below the analytical detection limit (LOD) and became nondetectable either immediately after the O₃-AOP stage or within a short hydraulic retention time in the MBR (Azizi et al., 2022). These outcomes strongly support the critical role of advanced oxidation processes in treating trace organic micropollutants and minimizing the ecological toxicity of the final effluent.

The detection limit of parabens below can be attributed to their high reactivity toward the generation of hydroxyl radicals (•OH) during ozonation. Phenolic structures and ester bonds in paraben molecules are particularly susceptible to electrophilic attack, leading to rapid ring cleavage and subsequent mineralization (Nguyen et al., 2021). Unlike conventional biological systems, which often fail to effectively degrade PPCPs because of their low concentrations and resistance to biodegradation, O₃-AOP pretreatment effectively destroyed these biologically active compounds before they reached the MBR (Narayanan et al., 2023). Consequently, the rapid reduction in parabens may lower the risk of endocrine disruption in receiving water bodies, although specific bioassays are needed to confirm the actual reduction in estrogenic activity. Notably, the present study focused on monitoring the disappearance of target paraben compounds and did not identify transformation products generated during ozonation. Consequently, while paraben concentrations decreased to below the analytical detection limit, the formation and environmental fate of intermediate oxidation products require further investigation in future studies.

2) Treatment efficiency of the model for different wastewater types

2.1) Mixed pharmaceutical-resin wastewater

The mixed wastewater scenario comprised a combination of pharmaceutical and resin synthesis effluents with moderate-to-high organic loading. Influent COD concentrations ranged from 2,750 to 3,310 mg L⁻¹, with phenol concentrations between 477 and 527 mg L⁻¹. This type of wastewater can cause significant shock loading in conventional aerobic biological systems if not properly pretreated.

As shown in Figure 3a, the COD concentration decreased progressively throughout the treatment stage, following an exponential decay pattern. Immediately after the ozone contact column, the COD concentration

decreased by approximately 50–53%, from approximately 3,300 mg L⁻¹ to approximately 1,500 mg L⁻¹. This substantial reduction at the chemical oxidation stage is mechanistically significant. Rather than achieving extensive degradation, ozone primarily functions by cleaving complex aromatic structures and transforming recalcitrant organic molecules into simpler, more biodegradable aliphatic intermediates (Soto-Verjel et al., 2026; Zheng et al., 2025). This structural conversion, rather than direct combustion, played a key role in enhancing biodegradability and reducing toxicity to the microbial community in the downstream MBR. As a result, the organic shock load to the biological stage was substantially mitigated, preventing potential sludge bulking or microbial inhibition.

In the MBR stage, the treatment performance remained stable. After a hydraulic retention time of 6 hours, the final effluent COD concentration stabilized at 113–128 mg L⁻¹, corresponding to an overall removal efficiency exceeding 96%. Although the system performed effectively from a technical standpoint, the presence of residual COD in the permeate indicates that a fraction of organic compounds remained resistant to both oxidation and biodegradation (Strotmann et al., 2026). To comply with increasingly stringent discharge standards, additional polishing steps, such as activated carbon adsorption, may be needed to further reduce the concentration of these recalcitrant organics.

Phenol removal was highly effective, achieving an overall efficiency of approximately 99%, with the concentration decreasing from approximately 500 mg L⁻¹ to 5–7 mg L⁻¹ in the final effluent. These results validate the effectiveness of ozonation in disrupting the stable aromatic ring of phenol. Moreover, parabens were reduced to concentrations below the analytical detection limit and were consequently reported as nondetectable (ND). Given the high reactivity of parabens toward hydroxyl radicals, their rapid, below-detection-limit removal further demonstrates the advantage of O₃-AOPs in treating wastewater containing trace amounts of biologically active micropollutants (Adeoye et al., 2024; Yacouba et al., 2021). The removal of such compounds not only satisfies regulatory requirements but also minimizes potential endocrine-disrupting risks to aquatic ecosystems.

2.2) High-strength pharmaceutical wastewater

High-strength pharmaceutical wastewater was the most challenging condition, with influent COD concentrations of up to 4,750 mg L⁻¹. This wastewater typically contains high levels of organic solvents and complex polycyclic compounds generated from pharmaceutical synthesis and extraction processes.

As presented in Figure 3b and 3c, a dramatic reduction in pollutant concentrations occurred along the height of the ozone contact column. At 0.5 m from

the bottom, the COD concentration decreased from 4,750 mg L⁻¹ to approximately 2,375 mg L⁻¹, representing a 50% reduction within the first half of the column height. This rapid decline indicates that gas–liquid mass transfer and oxidative reactions occurred rapidly in the initial contact zone, where the concentration gradient between pollutants and ozone was highest (Khakzad et al., 2026). The reaction kinetics in this region appeared to follow pseudo-first-order or second-order behavior, strongly dependent on the reactant concentration (Blitz et al., 2023).

Notably, the COD concentration continued to decrease sharply in the upper half of the column, reaching approximately 133 mg L⁻¹ at the top (1.0 m). The abrupt reduction from more than 2,000 mg L⁻¹ to less than 150 mg L⁻¹ in the upper section can be explained by two concurrent mechanisms. After large organic molecules were cleaved in the lower section, the resulting smaller intermediates became more susceptible to further oxidation (Durand et al., 2025). Additionally, intensive gas sparging likely induced stripping, facilitating the removal of volatile intermediates from the liquid phase (Son and Choi, 2023).

After the MBR stage, the effluent COD concentration remained stable at 103–133 mg L⁻¹, corresponding to an overall removal efficiency of 97–98%. However, these values still exceeded the Column A limit under QCVN 40:2025/BTNMT, suggesting that dilution or effluent recirculation may be necessary to meet the most stringent discharge standards. Phenol was effectively reduced from 134–192 mg L⁻¹ in the influent to 1–2 mg L⁻¹ in the final effluent. The superior phenol removal in the mixed wastewater scenario compared with that in the single wastewater scenario can be attributed to the lower influent phenol concentration, which led to a higher ozone-to-phenol ratio and more favorable conditions for extensive degradation.

2.3) Resin synthesis wastewater

Resin synthesis wastewater posed the greatest challenge because of its extremely high phenol concentration, which ranged from 677 to 865 mg L⁻¹. At such levels, phenol acts as a strong cytotoxin capable of disrupting microbial cell membranes and completely inhibiting biological activity in the MBR if not properly pretreated.

As shown in Figure 3c, the phenol concentration decreased progressively along the ozone column. After the full ozone treatment, the concentration decreased from approximately 865 mg L⁻¹ to approximately 300 mg L⁻¹. At specific sampling points, the phenol concentration sharply decreased to 187 mg L⁻¹ at 0.5 m and further to 85 mg L⁻¹ at 0.75 m. This reduction below the typical inhibitory threshold (<200 mg L⁻¹) before the MBR was entered was critical for maintaining microbial viability and activity during the biological stage.

In the final MBR effluent, the phenol concentration ranged from 9 to 13 mg L⁻¹, corresponding to an overall removal efficiency exceeding 98.5%. However, for wastewaters with exceptionally high phenol levels, a single-pass treatment may not be sufficient to achieve very low residual concentrations. An extended reaction time or increased ozone dosage may be required for further optimization.

Interestingly, COD removal in this scenario was the most favorable among the three wastewaters, with final effluent concentrations of 67–94 mg L⁻¹. One run even reached 67 mg L⁻¹, which is within the column B limit (90 mg L⁻¹) of QCVN 40:2025/BTNMT. This superior COD performance can be explained by the fact that the organic content in the resin wastewater was predominantly composed of phenol and its derivatives (Chruszcz-Lipska et al., 2025; da Silva et al., 2024). Once phenol was effectively degraded, a large portion of the COD was simultaneously removed.

A laboratory-scale hybrid coagulation–sedimentation/O₃-AOP/MBR system demonstrated suitable treatment performance for industrial wastewaters containing high concentrations of recalcitrant organic pollutants (Xiangyu et al., 2025). Across all three challenging wastewater scenarios, the system consistently achieved COD removal efficiencies exceeding 95% and effectively eliminated toxic phenolic compounds and trace micro-pollutants, such as parabens. These results demonstrate the highly complementary performance of combining chemical oxidation with biological degradation and membrane separation in a single integrated treatment train (Oller et al., 2010).

3) Spatial attenuation profile of pollutant degradation inside the ozone contact column

The spatial variation in pollutant concentrations along the height of the ozone contact column was examined to better understand the distribution of reaction zones and the dominant mechanisms occurring at different stages of the oxidation process. Attention was focused on the high-strength pharmaceutical wastewater, which had the highest influent COD concentration (4,750 mg L⁻¹). Samples collected at 0.5 m, 0.75 m, and 1.0 m from the bottom of the column were used to construct the spatial profile shown in Figure 4.

In the lower section of the column (0–0.5 m), the COD concentration decreased from 4,750 mg L⁻¹ to 2,375 mg L⁻¹. This corresponded to a removal efficiency of 50% within the first half of the column height. The observed reduction in this region was comparable to the value estimated using a pseudo-first-order kinetic model, suggesting that hydroxyl radical reactions proceeded rapidly under the high-concentration gradient conditions near the gas–liquid interface at the bottom of the column.

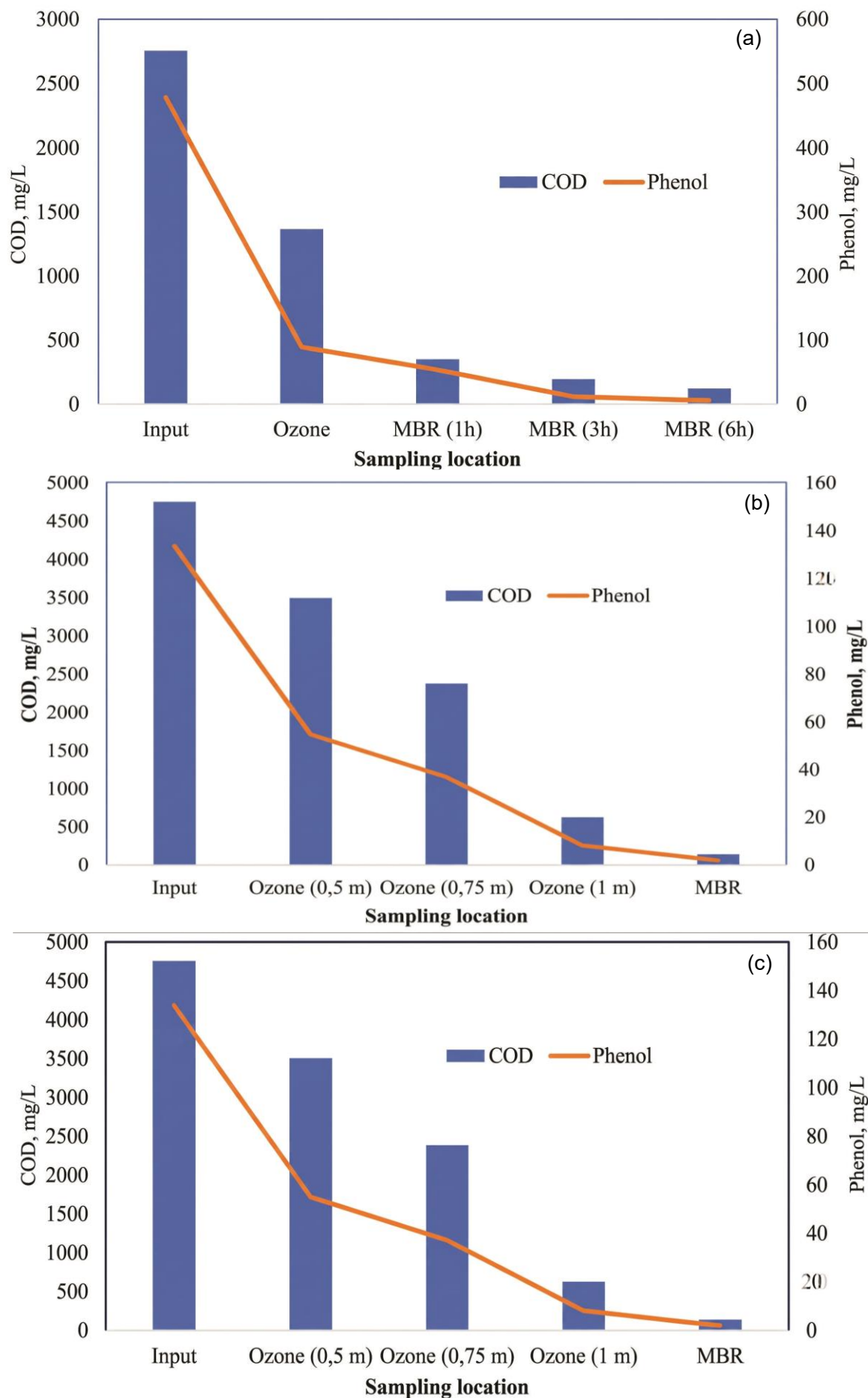


Figure 3 Removal profiles of bulk organic matter (COD) and target micropollutants (phenol and parabens) across successive treatment stages of the integrated hybrid system.

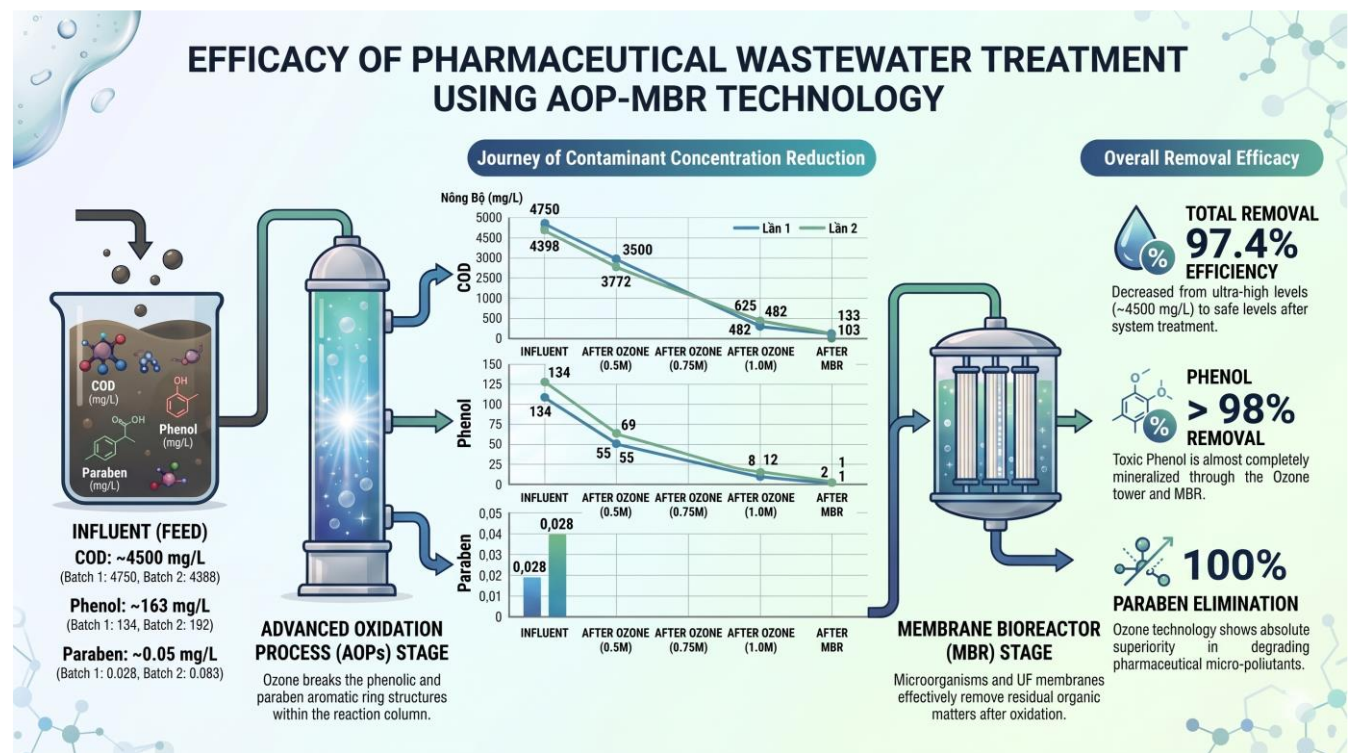


Figure 4 Spatial attenuation profiles of COD and phenol degradation along the vertical axis of the ozone contact column: Comparison between experimental values and the exponential spatial decay model.

Table 1 Comparison between experimental COD removal efficiencies and those predicted by the exponential spatial decay model at different heights of the ozone contact column

Column height (m)	COD (mg L ⁻¹)	Experimental removal efficiency (%)	Predicted baseline removal efficiency (%)
0.0 (Influent)	4,750	0.00	0.00
0.5	2,375	50.00	50.00 (Calibrated)
0.75	625	86.84	64.65
1.0 (Top)	133	97.20	75.00

Beyond 0.5 m, the rate of COD reduction continued, but the relationship between the experimental observations and the predicted baseline changed. At 0.75 m, the experimental removal efficiency reached 86.84%, while the exponential spatial decay model estimated 64.65%. At the top of the column (1.0 m), the experimental removal efficiency was 97.20%, compared with the baseline prediction of 75.00%. These differences are summarized in Table 1.

The increasing divergence between the experimental data and the baseline prediction in the upper section of the column suggests that additional removal mechanisms, beyond those accounted for by the exponential spatial decay model, may have contributed to the overall performance. One possible explanation is the physical stripping of partially oxidized intermediates facilitated by the continuous upward flow of ozone gas (Kato and Kansha, 2024).

As larger molecular structures were fragmented in the lower section, the resulting smaller compounds could have been more readily transferred from the liquid phase into the gas phase under the turbulent conditions created by gas sparging (Son and Seo, 2022).

However, because gas-phase compounds and dissolved ozone concentrations were not measured in the present study, the relative contributions of oxidation, stripping, mass-transfer effects, and hydrodynamic processes could not be quantitatively distinguished.

A comparable pattern was observed in the COD removal profile along an ozone-activated carbon column, as shown in Figure 5. In that system, the experimental removal efficiency closely followed the spatial decay baseline predictions up to approximately 0.5 m (which served as the calibration point). Above this height, however, the experimental curve differed from the predicted baseline, with the removal efficiency reaching 97.2% at 1.5 m compared with 75.0% estimated by the model. Although the two systems differ in target parameters and column configurations, the similarity in the spatial distribution of performance—where the experimental results increasingly exceed the baseline predictions in the upper stages—indicates that factors other than simple spatial decay profiles may influence overall removal in the upper portions of ozone-based contactors (Ju et al., 2026; Whelan and Kim, 2022).

Taken together, the spatial profiles in both systems suggest that the ozone contact column does not function uniformly along its height. The lower section appears to be predominantly influenced by rapid radical-driven oxidation under high concentration gradients, whereas the upper section may involve additional contributions from physical or surface-mediated processes (Kerner et al., 2021; Liu et al., 2020). These observations highlight the complexity of pollutant degradation in ozone contactors and suggest that both chemical and physical mechanisms should be considered when spatial performance data are interpreted and such systems are designed. Therefore, the proposed stripping contribution should be regarded as a plausible mechanistic hypothesis rather than direct experimental evidence.

4) Concentration reduction mechanisms for phenol and paraben

Industrial wastewater treatment must address not only high organic loading but also the presence of cytotoxic compounds and endocrine-disrupting chemicals (EDCs). The experimental results obtained from pharmaceutical and resin synthesis wastewater demonstrated the effective removal of target pollutants by the integrated O₃-AOP/MBR system.

4.1) O₃-AOP barrier mechanism for phenol

Phenol is a strong cytotoxin that can disrupt microbial cell membranes and inhibit biological activity at elevated concentrations. In the resin synthesis wastewater, the influent phenol concentration reached 865 mg L⁻¹, a level expected to severely impair MBR performance if it is introduced without pretreatment. As shown in Figure 6 (Chart 1), the O₃-AOP stage functioned as an effective “barrier” by rapidly reducing the phenol concentration in the lower section of the ozone contact column. In the resin wastewater, the phenol concentration decreased sharply from 865 mg L⁻¹ to 302 mg L⁻¹ at 0.5 m and further to 187 mg L⁻¹ at 0.75 m, falling below the typical biological inhibition threshold (approximately 200 mg L⁻¹). This reduction allowed the microbial community in the MBR to remain functional and to continue degrading the remaining phenol, resulting in final effluent concentrations of 9–13 mg L⁻¹.

The protective effect of the O₃-AOP stage was less critical but still evident in pharmaceutical wastewater, where influent phenol concentrations were substantially lower (134–192 mg L⁻¹). In this case, the oxidation process achieved a high degree of mineralization, resulting in final effluent phenol concentrations of only 1–2 mg L⁻¹ after the MBR stage. The difference in residual phenol between the two wastewaters suggests that the effectiveness of mineralization within a single-pass O₃-AOP system is influenced by the initial phenol loading and the available ozone dosage.

COD Removal Efficiency as a Function of Ozone Column Height: Experimental vs. Theoretical Model

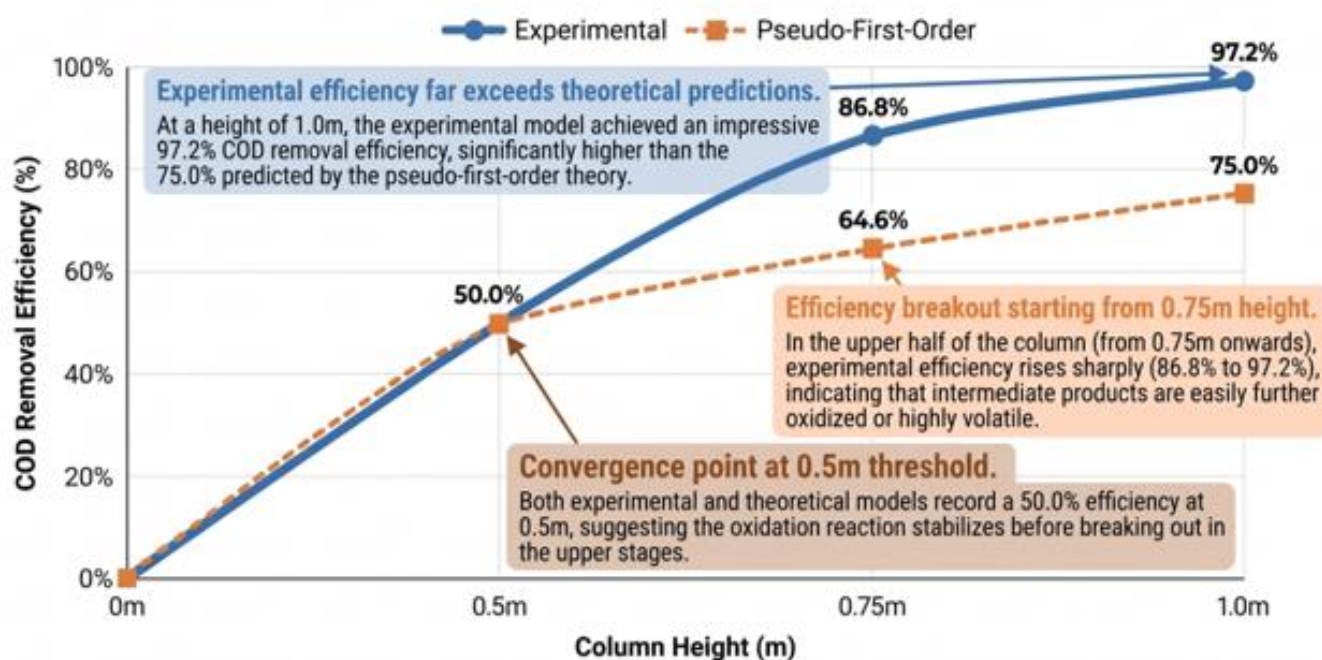


Figure 5 Comparison of spatial performance profiles and divergence from the exponential spatial decay model between the current ozone contact column and a reference ozone-activated carbon contractor.

4.2) Ultrafast oxidation mechanism for parabens

Unlike phenol, parabens are typically present at trace concentrations but are recognized as potent endocrine-disrupting compounds. Conventional biological treatment processes generally exhibit a limited ability to remove such micropollutants because of their low concentrations and resistance to biodegradation. As illustrated in Figure 6 (Chart 2), the O₃-AOP stage demonstrated exceptionally high reactivity toward parabens. In pharmaceutical wastewater, where influent paraben concentrations were very low (0.028–0.083 mg L⁻¹), paraben was completely removed to nondetectable levels immediately after the initial ozone contact stage. Even in the more challenging resin synthesis wastewater, where the influent paraben concentration ranged from 0.736 to 1.195 mg L⁻¹, the concentration sharply decreased to 0.121–0.192 mg L⁻¹ at 0.5 m and became undetectable from 0.75 m onward.

This rapid elimination is consistent with the high susceptibility of the phenolic ring and ester group of paraben to attack by hydroxyl radicals (Cuerda-Correa et al., 2016). The nonselective nature of •OH allows effective degradation across a wide range of influent concentrations, from trace levels to relatively high micropollutant loads (Lee et al., 2026). Consequently, parabens were consistently reduced to nondetectable levels in the final effluent of both wastewater scenarios, substantially lowering the potential for endocrine disruption in receiving water bodies.

4.3) Implications for the hybrid treatment system

The results presented in Figure 6 highlight the complementary roles of the O₃-AOP and MBR stages within the hybrid system. Ozone-based pretreatment effectively reduced acute toxicity associated with high phenol concentrations to levels compatible with those associated with biological treatment while simultaneously achieving rapid and complete removal of trace parabens (Xiangyu et al., 2025). This sequential action supports the multiple-barrier approach to advanced wastewater treatment, which combines chemical oxidation and biological processes to address both bulk organic pollutants and emerging micropollutants (Yacouba et al., 2021). The final effluent, characterized by substantially reduced phenol levels and nondetectable parabens, suggests a substantial reduction in the concentrations of target compounds associated with conventional and emerging toxicological concerns. However, dedicated toxicity assays would be needed to confirm a reduction in actual risk.

Conclusions

This study demonstrated the effectiveness of a laboratory-scale hybrid coagulation–sedimentation/O₃-AOP/MBR system for treating high-strength wastewater from pharmaceutical and resin synthesis processes containing persistent organic pollutants and toxic micropollutants. Across three representative wastewater scenarios, the hybrid system consistently achieved COD removal efficiencies above 95%, with the highest performance (97.4%) recorded in high-strength pharmaceutical wastewater. Phenol was effectively degraded to low residual concentrations (1–13 mg L⁻¹), while paraben concentrations decreased to below the analytical detection limit (LOD) and were subsequently reported as nondetectable (ND) in all cases.

Spatial kinetic profiling along the ozone contact column revealed that pollutant degradation occurred through two distinct mechanisms. In the lower section of the column, rapid oxidation followed pseudo-first-order kinetics at high concentrations. In the upper section, additional removal was attributed to the physical stripping effect induced by continuous gas sparging, resulting in significantly higher experimental removal efficiencies than those predicted by the kinetic model alone. The O₃-AOP stage played a critical protective role by reducing high phenol concentrations below the biological inhibition threshold, thereby enabling stable operation of the downstream MBR.

The results confirm that the integrated hybrid configuration provides complementary benefits by combining chemical oxidation for molecular cleavage and concentration reduction with biological degradation and membrane separation. This multiple-barrier approach effectively removed bulk organic loading and target trace pollutants, thereby minimizing the discharge of potentially toxic and endocrine-disrupting chemicals into the effluent. The final effluent quality approached or met the column B limits in QCVN 40:2025/BTNMT, indicating the system's practical potential for treating complex industrial wastewater. While substantial reductions in phenol and paraben concentrations indicate improved effluent quality, dedicated biological toxicity assays and carbon mass-balance analyses are needed to directly verify concentration reduction, mineralization, and ecological risk reduction. Future research should focus on optimizing the ozone dosage and hydraulic retention time for wastewater with extremely high phenol concentrations, evaluating changes in biodegradability and membrane fouling behavior, and evaluating system performance using real industrial wastewater.

ANALYSIS OF STRESS MECHANISMS FOR PHENOL AND PARABEN IN A HYBRID OZONE-MBR SYSTEM

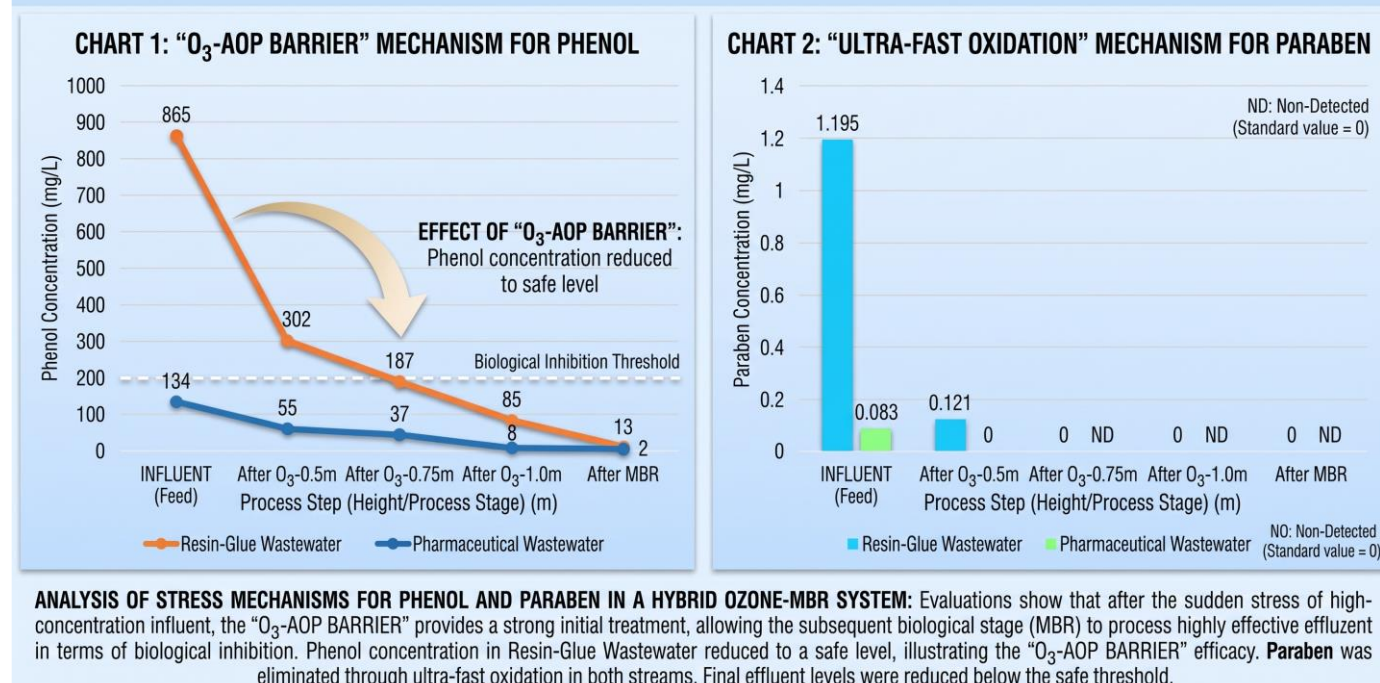


Figure 6 Schematic representation of the protective barrier role of the O₃-AOP stage in preventing biological inhibition and safeguarding the downstream MBR microbial community.

Data availability statement

This study received financial and technical support from the project "Establishment of laboratory-scale models for environmental pollution control, waste management, and their dissemination and replication for environmental protection".

Data availability statement

Information and data used in the study will be disclosed upon request.

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Conflicts of interest

The authors declare that there are no conflicts of interest in competing financial or personal relationships that could have appeared to influence the work reported in this work.

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